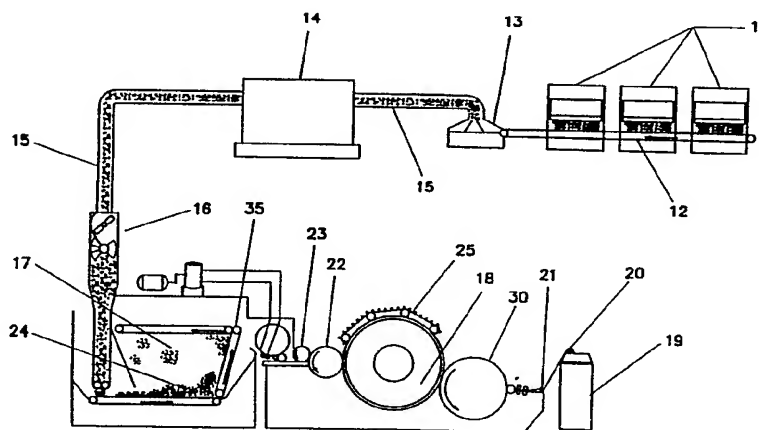


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<b>(21) International Application Number:</b> PCT/US91/01561 <b>(22) International Filing Date:</b> 6 March 1991 (06.03.91)  <b>(30) Priority data:</b> 493,178 14 March 1990 (14.03.90) US  <b>(71) Applicant:</b> ALLIED-SIGNAL INC. [US/US]; Law Department (C.A. McNally), P.O. Box 2245R, Morristown, NJ 07962-2245 (US).  <b>(72) Inventors:</b> KIRKLAND, Kevin, Mark ; 2908-2A East Stonyhill Court, Richmond, VA 23235 (US). WEBER, Charles, Paul, Jr. ; 1104 Martha Drive, Monroe, NC 28110 (US).		<b>(74) Agent:</b> ROONEY, Gerard, P.; Allied-Signal Inc., Law Department (C.A. McNally), P.O. Box 2245R, Morristown, NJ 07962-2245 (US).  <b>(81) Designated States:</b> AT (European patent), BE (European patent), CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>

**(54) Title:** SPINNING OF HIGH MOLECULAR WEIGHT POLYETHYLENE FIBER AND THE RESULTING SPUN FIBER**(57) Abstract**

A method of spinning discontinuous lengths (staple) of a high molecular weight (over 150,000) polyethylene fiber, and the resulting spun fiber, are disclosed. The method comprises blending a sufficient amount of discontinuous lengths of any other fiber (as a carrier) with the high molecular weight polyethylene fiber lengths to permit spinning. The resulting spun yarn preferably comprises about 5 to 95 weight percent discontinuous lengths of the high molecular weight polyethylene fiber which is characterized by a denier per filament of 20 or under and an ultimate elongation of 8 % or under at room temperature, and about 5 to 95 weight percent discontinuous lengths of the other fiber which is characterized by a denier per filament of 20 or under and which is either permanently crimped or has a memory. The carrier fiber or mixtures of carrier fibers may be chosen to achieve specific properties in the spun fiber: e.g., polyester or cotton for hand; pigmented polypropylene for color; aramids and polybenzimidazoles (PBI) for flame resistance; and carbon for chemical absorbance or resistance and for use in composites requiring strength and toughness. The spun fiber has utility in composites, industrial, medical and/or apparel applications.

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1  
SPINNING OF HIGH MOLECULAR WEIGHT POLYETHYLENE  
FIBER AND THE RESULTING SPUN FIBER  
BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to the spinning of staple fibers into yarn and to the resulting spun yarn. More particularly, it relates to the spinning of high molecular weight polyethylene staple fiber and to modifications to conventional spinning technology necessary to spin the high molecular weight polyethylene fiber.

2. The Prior Art

The spinning of staple (discontinuous lengths) fibers has been practiced for centuries. Natural fibers are of discontinuous lengths and require spinning to form a continuous yarn. Synthetic continuous filament fibers are converted into staple fiber and spun for a variety of reasons. One such reason is to improve the hand or appearance of the yarn. Another reason is to make a good yarn less expensively since the spinning process permits the use of some fiber which would have been wasted.

With the advent of the high molecular weight polyethylene continuous filament fibers which are produced by solution spinning, such as SPECTRA high strength polyethylene fiber, it was only a matter of time before an attempt was made to cut the continuous filament fiber into staple and spin the staple fiber a fiber into yarn. When the attempt was made, it was not possible to card the fiber since the fiber batt was so slippery that it could not be moved by the conventional feed roll to the card. Furthermore, when an attempt was made to overcome the slipperiness problem by crimping the fiber, it was found that the fiber would not hold crimp. Another reason to attempt crimping was that the high strength polyethylene fibers would not spring back from being compressed during normal spinning processing, which is contrary to experience with other fibers. Until this invention that problem was not solved.

The present invention was developed to overcome these problems.

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BRIEF DESCRIPTION OF THE INVENTION

This invention is a spun yarn, comprising discontinuous lengths of a high molecular weight polyethylene fiber and a sufficient amount of discontinuous lengths of any other fiber to permit spinning a blend of the fiber lengths.

The preferred molecular weight of the polyethylene is greater than about 300,000, and more preferred is the molecular weight of greater than about 500,000.

The preferred other fiber has memory. By memory is meant ability to return to original shape after distortion. For example a straightened, formerly crimped fiber that will return to its original crimped state.

The preferred other fiber has a denier per filament of no greater than 20, more preferably the other fiber has a denier per filament of no greater than 3.

The preferred discontinuous lengths of the polyethylene fiber and the other fiber are no longer than about 14 inches, more preferably the discontinuous lengths of the polyethylene fiber and the other fiber have a length ranging from about 0.75 to 14 inches.

Preferably, the polyethylene fiber forms about 5 to 95% by weight of the spun fiber, more preferably, the polyethylene fiber forms about 5 to 60% by weight of the spun fiber.

Preferably, the polyethylene fiber has an ultimate elongation of less than about 8% at room temperature, more preferably about 2 to 4.5%. Preferably the polyethylene fiber has a denier per filament of no greater than about 20, more preferably about 1 to 15, and most preferably, about 1 to 10. Even more preferably the polyethylene fiber has a denier per filament of about 1 to 3.

The preferred other fiber is selected from the group consisting of polyester fiber, polyamide fiber, cotton fiber, wool fiber, rayon fiber, polypropylene

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fiber, aramid fiber, lower molecular weight polyethylene fibers, carbon fiber, and mixtures thereof.

5 It is preferred that the other fiber forms about 5 to 95% by weight of the spun fiber and is characterized by having memory, a denier per filament of no more than about 20, and discontinuous lengths of no more than about 14 inches.

10 It is preferred that polyethylene fiber forms about 5 to 95% by weight of the spun fiber and is characterized by an elongation of no more than about 8% at room temperature, a denier per filament of no more than about 20, and discontinuous lengths of no more than about 14 inches.

15 It is more preferred that the other fiber is an aramid fiber which forms no more than about 50% by weight of the spun fiber.

The preferred aramid other fiber is poly(p-phenylene terephthalamide).

Use of lower price other fiber can create lower cost yarn blends.

20 This invention is also an article made from the spun yarn of the spun fiber described above, preferably the article made from the spun fiber of about 50% aramid.

25 This invention is also a method of spinning discontinuous lengths of a high molecular weight polyethylene fiber, comprising blending a sufficient amount of discontinuous lengths of any other fiber to permit spinning of a blend of the fiber lengths.

30 The preferred and more preferred molecular weights of the polyethylene are given above, along with the preferred, more preferred percent by weight blends, fiber weights, other fiber types, elongations, etc.

35 This invention is also an apparatus for moving a fiber batt of at least about 50 weight percent high molecular weight polyethylene fiber, comprising rotating means, said rotating means having a surface which rotates

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into contact with said fiber batt, said surface being modified across at least a portion thereof to increase the friction between the surface and the fiber batt so that the batt moves when contacted by the rotating surface.

5 It is preferred that the surface be modified by having an adhesive across said portion, a double-backed tape across said portion, or fabric capable of increasing friction or engaging the fiber (such as a Velcro tape or Emory cloth) attached to said portion.

10 The surface also can be modified by roughening to a surface of roughness of similar to 800 grit sandpaper or more coarse.

#### BRIEF DESCRIPTION OF THE DRAWINGS

In the accompanying drawings,

15 FIGURE 1 is a schematic overview of the prior art cotton carding process for sliver production;

FIGURE 2 is a detail of the card from FIGURE 1;

FIGURE 3 is a detail of the feed to the card, with emphasis on feed to the lickering;

20 FIGURE 4 is a detail of the modified feed roll 23;

FIGURE 5 has various views of trumpet 21.

25 FIGURE 5A is an isometric view of a prior art trumpet shown in section in FIGURE 5B; and FIGURE 5C is a section of a modified trumpet, showing the enlarged exit opening. FIGURE 5D is an end view.

#### DETAILED DESCRIPTION OF THE INVENTION

30 In the accompanying drawings, like numbers refer to like apparatus. The process which is depicted and which was used in the examples which follow was the cotton system of spinning yarn. For more detail on this system, reference may be had to Textile Processing Vol. I by John H. Marvin, (1973) - South Carolina State Dept. of Education Office of Vocational Education; and Textiles: Fiber to Fabric by M.D. Potter and B.P. Corbman (1975) - McGraw-Hill Inc., see Chpt. 3 pp 35 to 71.

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With reference to FIGURE 1, discontinuous fiber lengths, here staple, are placed by an operator into the backs of a plurality of hoppers 11. Inside each hopper 11, the individual mass of fibers is broken into smaller tufts by spiked conveying aprons (not shown) which then feed the tufts onto conveyor belt 12 to mix with tufts of fibers from other hoppers 11. Conveyor belt 12 then feeds the mixture of fibers into a blending unit 13 where they are further broken into smaller elements and further mixed. From unit 13 the fibers are transported through pipe 15 via Whitin Axi-Flo Unit 14 which is a pneumatic system, to pneumatic distributor 16. Distributor 16 further opens and fluffs the fibrous mass and then meters the fiber into the CMC Evenfeed Unit 17 which begins to form the fiber batt 35 from fiber 24 (referred to as a fiber lap in the art). With reference to Fig. 1, Fig. 2, Fig. 3 and FIGURE 5, fiber batt 35 is fed via feed plate 37 in association with feed roll 23 to lickerin 22 which has a plurality of wires 50 (card cloth) which rotate against batt 35 to pull and align discrete fibers from batt 35 and transfer them to the card cloth 50 covering the card cylinder 33 where they are further oriented and worked (brushed/combed) by the carding plates or flats 25. Doffer 30 is also covered with a card cloth, removes the accumulating sheet of oriented fibers from card 18 and carries the sheet of fibers to a removal point where it can be removed by a comb 28. The sheet of fibers is then fed through a trumpet 21 which forces the sheet to neck down for passage through a circular aperture 54. From aperture 54, the sliver 20 (necked down fiber sheet) is piddled into a take-up can 19 where it is loosely coiled.

The modification to feed roll 23 is shown in Fig. 4; double-sided adhesive tape 52 was spiralled around roll 23, as shown. Without use of an adhesive such as the tape, or another surface modification the feed roll does not function properly when high strength,

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high molecular weight polyethylene staple fiber is being processed, especially when blends over about 50% high molecular weight polyethylene are being processed. Without modification, the batt of fibers just slips and backs up, forms clumps and therefore the machine must be stopped because the fiber can not be carded.

Figs. 2,3,4 and 5 provide more detail regarding the prior art process showing feed roll 23 and surrounding members in greater detail.

Fig. 2 shows feed plate 37, card cylinder 33 screen 38, having wire 50 protruding, lickerin screen 40, backplate 34, spiral brush 31 for flats 25, front plate 29. Calender rolls 27 convey the sliver 20 from trumpet 21 to sliver can 19 on holder 26. Clearer 43, clears feed roll 23. Lap guide 45 guides the lap to feed roll 23. Mote knives 48 cut away motes which may accumulate from the lap.

Fig. 5 shows trumpet 21 in various aspects. Figure 5A is an isometric view and Fig. 5B is a cross section showing outlet opening 54 while 5D shows end view of trumpet 21 having outer perimeter 55 and outlet 54. Fig. 5C show the enlarged outlet opening 57, necessary for production of the spun fiber of this invention. For further reference to the general discussion which follows, refer to Textile Processing Vol.I, supra.

As previously indicated, when an attempt was made to spin staple high molecular weight polyethylene fiber alone with the system depicted in the drawing figures, feed roll 23 could not move batt 35 forward to the lickerin. Two modifications were made. The fiber was blended with other fiber, to be discussed in more detail below, and the feed roll 23 was modified as shown in FIGURE 4 with a spiral of double-backed masking tape on its surface that rotates into contact with batt 35 to permit it to move batt 35 forward. Anything that can be put on the feed roll 23 to enhance the frictional characteristics between its surface and batt 35 is



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acceptable, and it may be coextensive with the roll surface or distributed over a portion thereof as long as the batt 18 can be moved by it. For example, Velcro - type tape, Emery cloth, a roughened surface, adhesive, double-backed tape or the like can be used.

5 Another problem was encountered at trumpet 21. Due to the inability of the fiber to hold crimp, it does not spring back after deformation. After the neck down of trumpet 21 aperture 28, the sliver 20 is like a rod and difficult to piddle. Therefore, trumpet 21 was bored  
10 out sufficiently to increase the size of the sliver 20 and to reduce or limit the compression of the sliver to the extent that it could be piddled (see FIGURE 5C).

Cans of the attenuated sliver are collected and transferred to a draw frame, in the accompanying examples  
15 a Saco Lowell four over five draw frame, where several individual slivers are blended and further attenuated/drawn.

The cans of attenuated, blended sliver are then transferred to a roving frame for further attenuation and  
20 a very low level of twist is inserted. The sliver is now ready for spinning and is taken-up on a bobbin which is the feeder package for the spinning frame.

The bobbins are hung on a spinning frame, preferably a ring spinning frame, where the slivers are  
25 again individually attenuated and spun into yarn by twisting. The spun yarn is then taken up for packaging or further processing.

U.S. Patent 4,457,985, hereby incorporated by reference, generally discusses high molecular weight  
30 polyethylene. In the case of polyethylene, suitable fibers are those of molecular weight of at least about 150,000, preferably at least about 300,000, more preferably at least about 500,000, most preferably in excess of about one million. Such extended chain  
35 polyethylene (ECPE) fibers may be grown in solution as described in U.S. Patent 4,137,394 or U.S. Patent

4,356,138, hereby incorporated by reference, or may be a filament spun from a solution to form a gel structure, as described in German Off. 3,004,699 and GB 2,051,667, and especially described in U.S. Patent 4,551,296, also hereby incorporated by reference. As used herein, the term polyethylene shall mean a predominantly linear polyethylene material that may contain minor amounts of chain branching or comonomers not exceeding 5 modifying units per 100 main chain carbon atoms, and that may also contain admixed therewith not more than about 50 weight percent of one or more polymeric additives such as alkene-1-polymers, in particular low density polyethylene, polypropylene or polybutylene, copolymers containing mono-olefins as primary monomers, oxidized polyolefins, graft polyolefin copolymers and polyoxymethylenes, or low molecular weight additives such as lubricants, colorants, fillers and the like which are commonly incorporated by reference. Depending upon the formation technique, the draw ratio and temperatures, and other conditions, a variety of properties can be imparted to these filaments. The tenacity of the filaments should be at least about 8 g/d, preferably in the range of from about 8 to 45 g/d, most preferably in the range of about 25 to 35 g/d/. Similarly, the tensile modulus of the filaments, as measured by an Instron tensile testing machine, is at least about 160 g/d, preferably in the range of about 150 to 3300 g/d, most preferably in the range of about 1,200 to 2,500 g/d. These highest values for tensile modulus and tenacity are generally obtainable only by employing solution grown or solution spun, gel filament processes.

The polyethylene fiber should preferably have an ultimate elongation by Instron tensile test of no greater than 8%, preferably in range of 2 to 4.5%, at room temperature, i.e., about 25° C. The denier per filament is preferably no greater than 20, more preferably 1 to 15, most preferably 1 to 10, even more preferably 1 to

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3. The smaller denier per filament fibers are desirable to use in spinning blends for apparel applications. The larger denier per filament fibers are better to use in spinning blends for cordage, ropes, or heavy fabric applications.

5           The amount of polyethylene fiber in the blend ranges from about 5 to 95% by weight, preferably about 20 to 80 % by weight as the primary fiber and about 5 to 40% by weight as the support fiber.

10           The carrier fiber may be any other fiber. i.e., anything except the high molecular weight polyethylene fiber just described. Exemplary fibers may be found listed in allowed U.S. Patent Application S.N. 62,998 filed July 13, 1987, hereby incorporated by reference. A partial listing would include the aramids, cottons,  
15           carbon, polyester, polyamides, wool, rayon, polypropylene, lower molecular weight polyethylenes, etc.

          The carrier fiber preferably should have the following characteristics: it should preferably be a fiber which has memory or is permanently crimped; it  
20           should preferably have a denier per filament of no greater than 20, more preferably 0.5 to 3 for apparel applications and preferably 3 to 20 for ropes, cordage, and heavy fabric applications.

          The minimum amount of carrier fiber needed is  
25           about 5% by weight of the spun fiber, and up to 95% by weight is acceptable. The preferred amount will range from about 20 to 50% by weight. The amount will be a function of the denier of the polyethylene fiber, e.g. one would need about 30% by weight carrier fiber to spin  
30           a 20 denier per filament polyethylene fiber, and would need less to spin a lower denier per filament polyethylene fiber.

          Both the carrier fiber and the polyethylene fiber are formed from discontinuous lengths of no greater  
35           than about 14 inches, preferably no longer than 2.25 inches in the cotton system (more preferably 0.75 to 2 inches).

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The particular blends of carrier fibers will depend on desired properties of the spun fiber. It is contemplated that a blend of polyester and/or cotton with the polyethylene will enhance hand; polypropylene can be blended for pigmentation; aramids, specifically Kevlar and Nomex can be included for flame resistance; polybenzimidazoles (PBI), available commercially from Hoechst-Celanese, can be included for flame resistance and enhanced comfort due to higher moisture regain; carbon for use in fiber for composites requiring strength and toughness. Activated carbon fiber could be used for chemical absorption protection.

#### EXAMPLES

##### EXAMPLE 1 (including comparative)

Using the apparatus of Fig. 1, a two-inch SPECTRA 900 fiber available from Allied-Signal Inc. is run on the apparatus, unsuccessfully. The fiber slips on the feed rolls, clumps up in the feed mechanism and the machine must be stopped. A second trial was attempted with a fiber blend of 70% of the SPECTRA 10 dpf fiber above and 30% 1.2 denier per filament (dpf) Kodel polyester 1.5 inch staple. The mixture of 70% Spectra 10 dpf two inch staple and 30% 1.2 dpf Kodel 1.5 inch polyester staple was prepared and loaded into the card feeder. Difficulty in carding the fiber mixture was encountered after about 30 minutes because the polyester fiber rolled on itself and formed small balls of fiber. The screen inside the Even Feed unit could not move the bat of Spectra fiber and the balls of polyester fiber to the card main cylinder. The fiber bat was probably too slippery to be moved by the feeder mechanism. The trial was terminated and the card feeder was cleaned.

The second trial was conducted using a blend by weight of 60% Spectra 900 10 dpf 2.0 inch staple fiber and 40% Kevlar 1.5 dpf 1.5 inch staple fiber. A length of double-backed adhesive tape was spirally wrapped around feed roll 23 as in Fig. 4. The difference in the two

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fibers dpf caused some problems with how well the mixture blended and carded. The problems were not serious. However, to optimize the process it may be desirable to have fibers which have similar dpf.

5 A total of 50 pounds of Spectra/Kevlar fiber blend was made. To control static electricity Stay Guard antistatic spray was purchased from a local grocery store. The anti static spray was added to the fiber in the opener as the fiber was being opened and blended. There were no static electricity problems.

10 The main problem encountered during carding was the loading of the card main cylinder by the Spectra fiber. This problem may be solved by using a fiber with a smaller dpf or development of a special fiber finish. There was also a large amount of Spectra fiber removed  
15 by the card flats. The card flats rotate against the main cylinder to help clean it. This problem may disappear if the cylinder loading problem can be solved.

The carded sliver was divided into six ends which were fed into a Saco Lowell 4 over 5 draw frame.  
20 The draw frame was running black coated rubber rolls. The black rolls seemed to minimize the tendency of the sliver to wrap. The draw frame further blended the carded sliver and reduced the sliver weight by about 50%.

The roving was produced on a Saco Lowell roving  
25 frame. No problems were encountered during the roving formation.

The roving was converted to spun yarn on a ring spinning frame. The speeds and conditions were varied to produce a yarn which had a specific cotton count. The  
30 first yarn count attempted was a 20's.

The spinning of the 20's yarn was very difficult because the yarn kept breaking. A calculation showed that because of the fiber diameters, the formation of a 20's count yarn was on the edge of being a physical  
35 impossibility. (Theoretically, at least 88 fibers are required in the cross section of a spun yarn. Because of

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the dpf of the Spectra fiber, it is impossible to have enough fiber in the spun yarn cross section and the yarn still be small enough to be a 20's.)

The two spun yarns produced were a 10's and a 5's cotton count. Both of these yarns spun easily. Some optimization will be necessary to achieve the best balance of physical properties in the finished yarn.

#### EXAMPLE 2

Example 1 was repeated loading the following fibers:

70% of 3 dpf SPECTRA \* 2" staple  
30% of 1.2 dpf Kodel polyester 1.5" staple  
Four spun yarns were successfully produced:  
10 cotton count single, 10 cotton count two-ply,  
20 cotton count single, 20 cotton count two-ply.

\*The SPECTRA fiber had a lower intrinsic viscosity of between about 6 and 8, estimated molecular weight about 700,000.

#### EXAMPLE 3 (Comparative)

An attempt to spin 100% SPECTRA fiber on the unmodified apparatus of Example 1 was unsuccessful, as was another attempt on a worsted spinning system.

#### SPECTRA / ARAMID BLEND SPUN YARN GLOVE

The article made from the blend of Spectra and Kevlar fiber from Example #1 exhibited good flame resistance and insulating properties. When the fabric (in this case a knitted glove) was exposed to a flame from a cigarette lighter the exposed polyethylene fibers on the fabric surface melted back into the fabric even with the aramid fiber surface. The fabric did not ignite or burn which is surprising because Spectra fiber is a polyolefin which will burn if it becomes hot enough. The inside of the glove remained cool. The Spectra fibers did not seem to melt on the side of the fabric opposite the flame. When an attempt to intentionally ignite the fabric made from the blend of Spectra and aramid fibers was made the fabric exhibited a self extinguishing

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behavior and did not readily support combustion.

(Surprising because olefin fibers are known to burn).

The Spectra fiber should improve the abrasion resistance of the aramid fiber (in the case of the glove) and allow the item to have a longer service life. The fabric made from the blend of fibers had a good hand and the frictional characteristics of the fabric surface were improved over an all Spectra fabric because the aramid fiber reduced the slickness of the surface. This improved frictional property would be desirable in a glove because of the necessity of keeping a good grip on objects being handled.

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## WE CLAIM:

1. A spun yarn, comprising discontinuous lengths of a high molecular weight polyethylene fiber and a sufficient amount of discontinuous lengths of any other fiber to permit spinning a blend of the fiber lengths  
5 wherein the molecular weight of the polyethylene is greater than about 300,000.
2. The yarn of claim 1 wherein the other fiber has a denier per filament of no greater than about 20.
3. The yarn of claim 1 wherein the discontinuous  
10 lengths of the polyethylene fiber and the other fiber are no longer than about 14 inches.
4. The yarn of claim 1 wherein the other fiber is selected from the group consisting of polyester fiber, polyamide fiber, cotton fiber, wool fiber, rayon fiber,  
15 polypropylene fiber, aramid fiber, lower molecular weight polyethylene fibers, carbon fiber, and mixtures thereof.
5. The yarn of claim 2 wherein the other fiber is an aramid fiber which forms no more than about 50% by weight of the spun fiber.
- 20 6. The yarn of claim 2 wherein the other fiber is poly(p-phenylene terephthalamide).
7. An article made from the spun yarn of claim 3.
8. An article made from the spun yarn of claim 1.
9. A method of spinning discontinuous lengths of  
25 a high molecular weight polyethylene fiber, comprising blending a sufficient amount of discontinuous lengths of any other fiber to permit spinning of a blend of the fiber lengths wherein the molecular weight of the polyethylene is greater than about 300,000 and the polyethylene fiber forms  
30 about 5 to 95% by weight of the blend.
10. The method of claim 9 wherein the other fiber is selected from the group consisting of polyester fiber, polyamide fiber, cotton fiber, wool fiber, rayon fiber, polypropylene fiber, aramid fiber, lower molecular weight  
35 polyethylene fibers, carbon fiber, and mixtures thereof.
11. Apparatus for moving a fiber batt of at least about 50 weight percent high molecular weight polyethylene fiber, comprising rotating means, said rotating means



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having a surface which rotates into contact with said fiber batt, said surface being modified across at least a portion thereof to increase the friction between the surface and the fiber batt so that the batt moves when contacted by the rotating surface.

- 5           12. The apparatus of claim 11 wherein the fiber batt is processed further on a card having trumpet to form the batt into a sliver, said trumpet being modified by having an enlarged exit opening.

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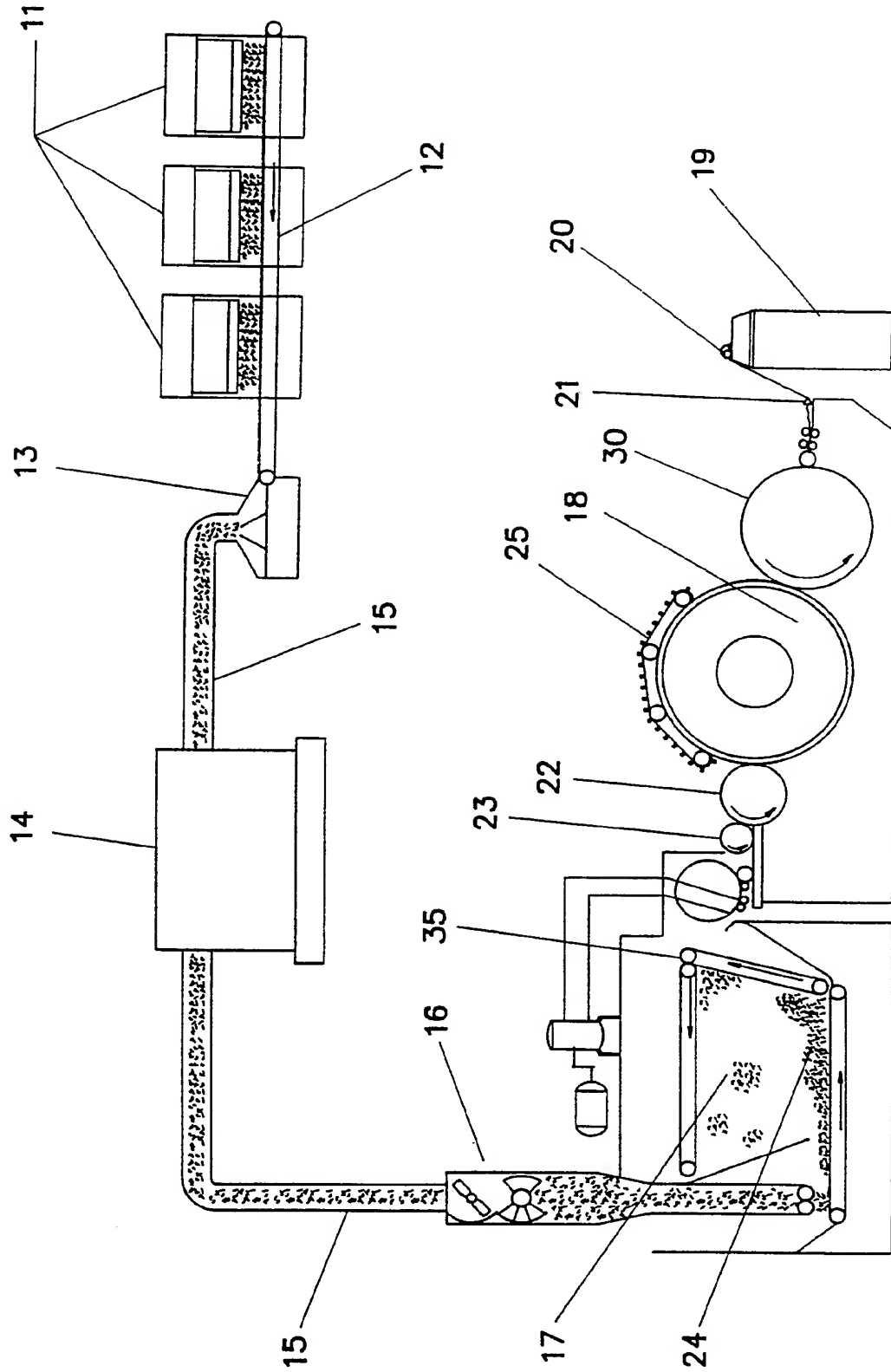


FIGURE 1  
PRIOR ART

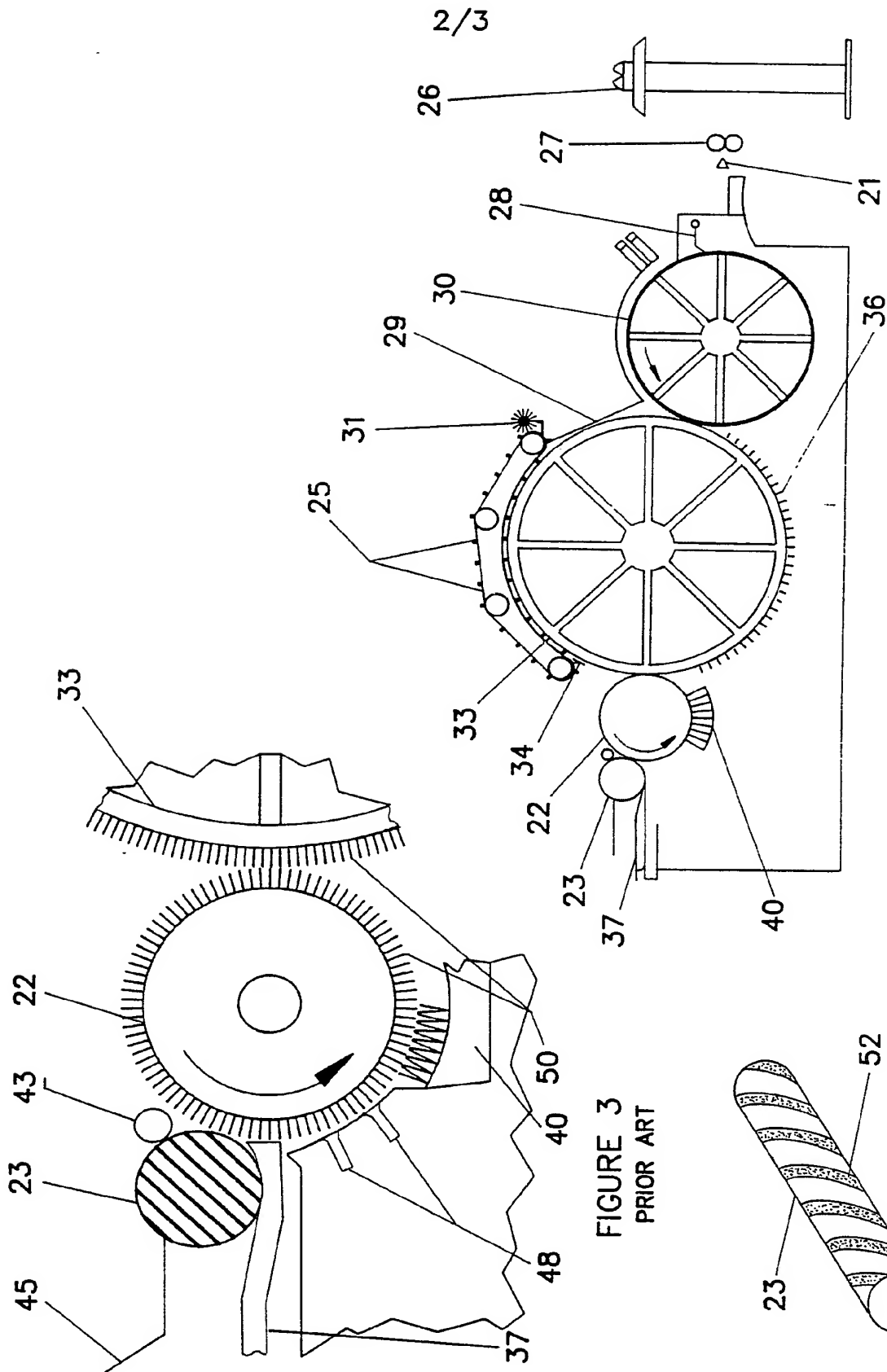


FIGURE 2  
PRIOR ART

**FIGURE 3**  
**PRIOR ART**

FIGURE 4

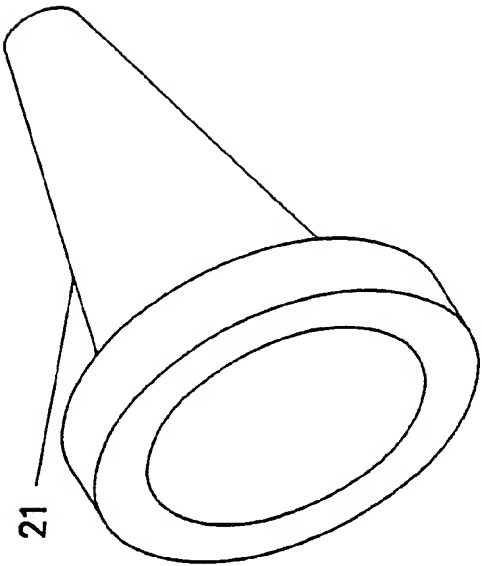


FIGURE 5A  
PRIOR ART

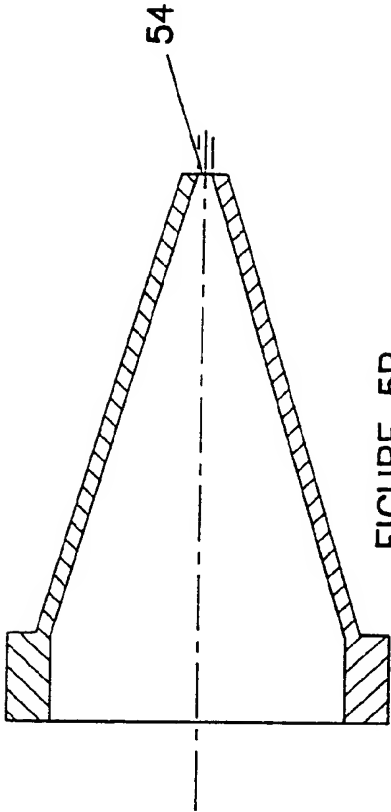


FIGURE 5B  
PRIOR ART

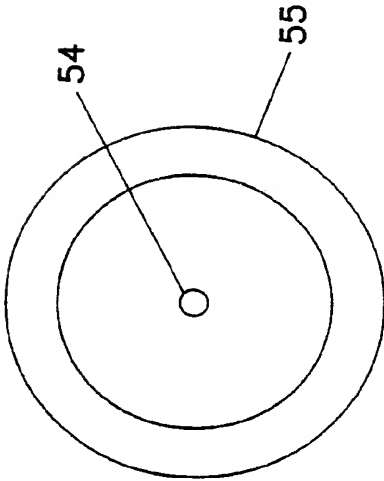


FIGURE 5D  
PRIOR ART

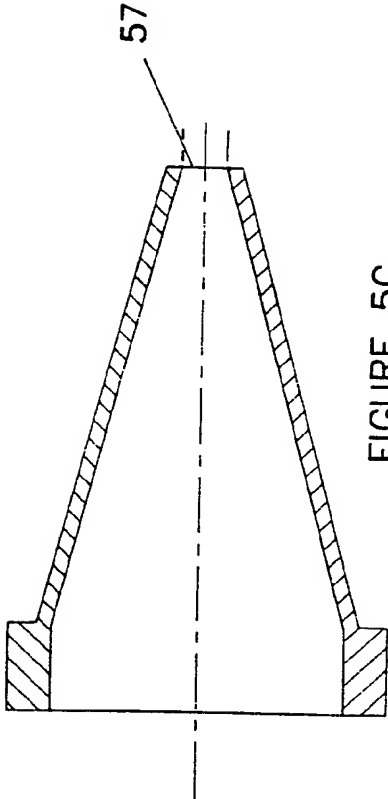


FIGURE 5C

# INTERNATIONAL SEARCH REPORT

International Application No PCT/US 91/01561

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)<sup>6</sup>

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: D 02 G 3/04, D 01 F 6/04

## II. FIELDS SEARCHED

Minimum Documentation Searched<sup>7</sup>

Classification System

Classification Symbols

IPC5

D 02 G; D 01 F

Documentation Searched other than Minimum Documentation  
to the Extent that such Documents are Included in Fields Searched<sup>8</sup>

## III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup>

Category \*

Citation of Document,<sup>11</sup> with indication, where appropriate, of the relevant passages<sup>12</sup>

Relevant to Claim No.<sup>13</sup>

A

EP, A1, 0310202 (STAMICARBON B.V.)  
5 April 1989,  
see the whole document

1-12

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A

GB, A, 2152543 (BERTRAND GOLDENSTEIN)  
7 August 1985,  
see the whole document

1-12

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A

US, A, 4457985 (GARY A. HARPELL ET AL.)  
3 July 1984,  
see the whole document

1-12

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### \* Special categories of cited documents:<sup>10</sup>

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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## IV. CERTIFICATION

Date of the Actual Completion of the International Search

18th June 1991

Date of Mailing of this International Search Report

30.07.91

International Searching Authority

EUROPEAN PATENT OFFICE

Signature of Authorized Officer

F.W. HECK



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**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO. PCT/US 91/01561**

SA 45948

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.  
The members are as contained in the European Patent Office EDP file on 29/05/91  
The European Patent office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A1- 0310202	05/04/89	JP-A- 1156538	20/06/89
GB-A- 2152543	07/08/85	NONE	
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For more details about this annex : see Official Journal of the European patent Office, No. 12/82